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Moisture Determination in Composite Materials Using Positron Lifetime Technique

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Moisture Determination in Composite Materials Using Positron Lifetime Technique

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SUMMARY

Fiber-reinforced resin-matrix composites reportedly suffer significant degradation in their mechanical properties when they are exposed to hot, moist environments for extended periods. Moisture weakens the fiber-matrix bond as well as the matrix shear strength. Despite the importance of measuring moisture content and its effects on composite material properties, not enough data are available on suitable nondestructive techniques for detecting and measuring moisture diffusion in resin-matrix composite materials. This paper addresses the problem of measuring the moisture content of such materials, using the positron lifetime technique.

INTRODUCTION

Low density and high strength of fiber-reinforced polymeric composites make them attractive candidates for lightweight structures in aerospace applications. However, several recent studies have shown that these materials pick up moisture when exposed to hot, moist environments for extended periods. (See refs. 1 to 5.) The equilibrium moisture pickup is a strong function of the temperature and the relative humidity of the ambient air at the material surface. (See refs. 6 to 8.) The presence of moisture in these materials degrades their mechanical properties. Water vapors migrate along the fiber-matrix interface, thereby weakening the fiber-matrix bond. Water also diffuses through the polymeric matrix itself and degrades the matrix-dominated properties of the composite materials. Furthermore, the daily absorption-desorption cycles experienced by the polymeric composites in service cause cyclic stress conditions in the outer layers of these materials and produce fatigue damage in their mechanical properties. The rate of moisture absorption in composites is controlled by the diffusivity of the matrix and the fiber, volume fractions of the fibers, and the orientation of the fibers with respect to the exposed surfaces. A proper understanding of the factors affecting moisture absorption is necessary before any effective steps can be taken to minimize the degrading effects of environmental moisture on the polymeric composites. Also necessary is the development of sensitive nondestructive techniques for measuring the moisture content in composite materials. Despite its importance, not enough data are available on suitable nondestructive techniques for detecting and measuring moisture diffusion in polymeric composites. Amongst the techniques that have been used for nondestructive measurement of moisture degradation in fiber-reinforced polymeric materials are ultrasonic methods, nuclear magnetic resonance pulse relaxation spectroscopy, and microwave techniques. Ultrasonic velocity measurement, coupled with moisture evolution analysis, offers the greatest versatility in providing the necessary nondestructive scanning capability for detection of environmental aging effects on large composite structures. (See refs. 9 to 11.) However, none of these techniques is suitable for simultaneous measurement of moisture content as well as its depth distribution in the test specimens. A new technique has been developed which has the potential of providing information on the moisture content as well as its depth

distribution in the specimen. This technique is based on the dependence of positron lifetime on the moisture content of the composite specimen. The positron lifetime technique of moisture determination and the results of the initial studies are described in the following sections.

MATERIALS

The following materials, or some combination of them, were used in the present study.

Narmco 5208 resin, manufactured by Narmco Materials, a subsidiary of Celanese Corporation

Thornel 300 graphite fibers (T300), manufactured by Union Carbide Corporation

Kevlar 49 aramid fibers, manufactured by E. I. du Pont de Nemours & Co., Inc.

HBRF-55A epoxy resin, supplied by Hercules Incorporated

Kapton polyimide resin, manufactured by E. I. du Pont de Nemours & Co., Inc.

HTS graphite fibers, manufactured by Hercules Incorporated

PMR-15, NASA-developed polyimide resin

Identification of commercial products in this report is to adequately describe the materials and does not constitute official endorsement, expressed or implied, of such products or manufacturers by the National Aeronautics and Space Administration.

THEORY

Before describing the positron lifetime technique and the initial results obtained so far, the basic phenomenon of positron annihilation in molecular substances is reviewed briefly. A more detailed account of positron annihilation in condensed matter can be found in references 12 and 13. A positron is the antiparticle of an electron. As such, it has the same mass as the electron but equal and opposite charge and magnetic moment. When a free positron encounters an electron, the two annihilate each other with a lifetime of 125 ps, releasing an energy of $2m_oc^2$, which is the total rest energy of the annihilating pair. When a positron enters a molecular medium, the following events take place:

(1) Energetic positrons injected into a condensed medium rapidly slow down to thermal energies by collisions with electrons and ions. After coming to thermal equilibrium, the positrons may annihilate with an electron from the surrounding medium into two 511-keV photons. The cross section for the 2-photon annihilation of a free positron with a stationary electron $\sigma(2\ \text{photons})$ was shown by Dirac (ref. 14) to be expressed as

$$\sigma(2\ \text{photons}) = \frac{\pi r_o^2}{\gamma + 1}\left\{\frac{\gamma^2 + 4\gamma + 1}{\gamma^2 - 1}\ln\left[\gamma + (\gamma^2 - 1)^{1/2}\right] - \frac{\gamma + 3}{(\gamma^2 - 1)^{1/2}}\right\} \qquad (1)$$

where

$$\gamma = \left(1 - \frac{v^2}{c^2}\right)^{-1/2}$$

v velocity of positron

c velocity of light

r_o classical electron radius, $\dfrac{e^2}{m_o c^2}$

At low positron energies (typically a few electron volts), an annihilation cross section inversely proportional to the positron velocity is obtained; that is,

$$\sigma(2\ \text{photons}) = \frac{\pi r_o^2 c}{v} \qquad (2)$$

This leads to an annihilation rate $\Gamma(2\ \text{photons})$ given by the following expression:

$$\Gamma(2\ \text{photons}) = \sigma(2\ \text{photons})\, v n_e = \pi r_o^2 c n_e \qquad (3)$$

The annihilation rate is clearly independent of the positron velocity and is directly proportional to the electron density n_e. Thus a measurement of the annihilation rate provides direct information about the electron density at the positron annihilation site.

(2) The positron, after having come to thermal equilibrium at the end of its range, may pair up with an electron to form a positronium atom.[1] These positronium atoms move in the free volume in the polymeric materials and exist in the following two forms:

(a) The singlet $\left(1\ ^1S_0\right)$ state or parapositronium. The lifetime of parapositronium for self-annihilation into two photons is equal to 125 ps, the same as for free positrons.

(b) The triplet $\left(1\ ^3S_1\right)$ state or orthopositronium. The orthopositronium atoms in vacuum would decay by 3-photon emission with a lifetime of about 140 ns. A competing mechanism, called pick-off annihilation (refs. 13, 15, and 16), causes orthopositronium to suffer a relatively faster 2-photon decay in condensed materials. In the pick-off process, the positron bound in an orthopositronium atom undergoes a 2-photon annihilation with a "foreign" electron having opposite spin. The lifetime for this process is typically from 2 to 4 ns with intensities comprising 10 to 30 percent of all annihilations.

The free-positron annihilation and parapositronium decay are not very interesting for the purpose of this investigation since they do not reflect the effects of electron distribution perturbed by the diffusing water. However, the pick-off annihilation of othropositronium atoms (delayed positron annihilation) is important since it includes the effects of positronium interaction with the surrounding medium and responds sensitively to the changes in the properties of the medium produced by the diffusing water. (See refs. 13, 15, and 16.) An alternative explanation for the delayed positron annihilation involves the trapping of positrons in the free volume of the composite system. When the free volume is reduced by the diffusion of water in the specimen, some of the positrons trapped there are forced into the region where "normal" electrons attached to the lattice molecules are present. This increases the chances of annihilation of the positrons, which reduces their lifetime. However, there is some experimental evidence (ref. 13) involving annihilation photon angular correlation which supports the formation of positronium atoms in free volume in polymers. Regardless of the mechanism responsible for delayed positron annihilation - whether pick-off by orthopositronium atoms or trapped positrons being forced into the excluded region - the lifetime is expected to show a correlation with the moisture content of the test sample.

EXPERIMENTAL PROCEDURE

Most positron lifetime studies make use of positron radioactive sources, which, by the emission of a simultaneous gamma ray, provide a "reference" time for the positron lifetime measurement. The time delay between the detection of this reference gamma ray in detector 1 at time t_1 and the subsequent positron

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[1]The formation of positronium atoms is energetically possible in polymeric materials. Experimental evidence (ref. 13), based on angular correlation of annihilation photons, supports the formation of positronium atoms in the polymeric materials.

annihilation photon detection in detector 2 at time t_2 is a measure of the positron lifetime in the medium where the positrons annihilate. Figure 1 is a schematic diagram of the fast-slow coincidence system used for positron lifetime measurement. The operation of this system has been described elsewhere (ref. 17), and only the essential details are briefly reviewed here. The fast "timing" channels include timing discriminators and a time-to-pulse height converter (TPHC) which produces signals with an amplitude proportional to the time interval $(t_2 - t_1)$ between the detection of the two events in detectors 1 and 2. The slow "energy" discriminating channels provide further identification of the pair of events between which the time interval is being measured. The lifetime spectrum consists of the number of events as a function of the time delay between their arrival at the respective detectors. The lifetime system resolution was approximately 450 ps, as measured with a Co^{60} source which emits two almost simultaneous (0.7-ps) gamma rays. A 10-μCi Na^{22} source ($Na^{22}Cl$) placed between two thin gold foils (2.4 mg/cm^2) was used as a positron emitter in the present study. (1 Ci = 3.7 $\times$ 10^{10} Bq.) The gold foils prevented contamination of the test samples with radioactive material. The emitted positrons have energies up to 0.54 MeV and can easily penetrate through the protective gold foils. The source assembly was sandwiched between two test composite coupons (25.4 $\times$ 25.4 $\times$ 3.2 mm^3) for measuring positron lifetime in them. The source-test coupon sandwich was wrapped in thin (0.025 mm) aluminum foil and sealed with vinyl tape to prevent moisture pickup/loss during the positron lifetime measurement. The source-target assembly was further sealed in a polyethylene bag and placed between the two detectors. A desiccant was included in the polyethylene bag for dry specimen measurements, whereas a water-saturated paper wick was included for the saturated specimen. A typical positron lifetime experiment required about 24 hours for a lifetime-measurement statistical accuracy of $\leq$1.5 percent. Lifetime measurements were made on dry, partially saturated (approximately uniform), and fully saturated test specimens. Completely dry specimens were prepared by heating the test samples to 120° C in a vacuum desiccator until the specimen weight became constant. Fully water-saturated specimens were prepared by boiling the test samples in distilled water for several days until there was no further increase in weight. Partially saturated (approximately uniform) samples were prepared by repeated boiling in water followed by drying in the desiccator until a predetermined amount of water was left in the sample. Partially saturated (uniform) samples in some of the later measurements were also prepared by suspending the specimen in a uniform-temperature enclosure containing a saturated solution of an appropriate salt at the bottom. The specimen was allowed to stand in this enclosure until there was no further weight gain. Some appropriate salts and their characteristics (ref. 18) are listed in table I.

Figure 2 shows a typical lifetime spectrum in dry HBRF-55A epoxy. Two prominent lifetime components are seen. The short-life component is associated with free-positron annihilation and parapositronium decay. The long-life component arises from pick-off annihilation of orthopositronium. As indicated previously, the long-life component shows sensitivity to the moisture content in the test specimen.

RESULTS

The experimentally observed positron lifetime spectra are the sum of at least two exponential components. These components have different lifetimes and intensities and, as indicated previously, result from different decay processes. The experimental data were fitted by the least-squares method, to a two-component expression of the following form:

$$n = n_1 e^{-\lambda_1 t} + n_2 e^{-\lambda_2 t} + b \tag{4}$$

where n is the total count in a channel on the right side of zero time, n_1 and n_2 are extrapolated zero-time intercepts for the two components, λ_1 and λ_2 are the respective decay constants, t is time, and b is constant chance coincidence background.

Since the long-life component is the crucial moisture-sensitive parameter of interest in this study, only the measured values of the long lifetime $\left(\tau_2 = \frac{1}{\lambda_2}\right)$ are considered. The results of moisture-induced reduction in long component lifetime for several composite samples (refs. 16 and 17) are summarized in figures 3 and 4. It is apparent that the positron lifetime decreases linearly with the moisture content of the specimen. The statistical accuracy of the lifetime data is about 1.5 percent, which is adequate to measure 10 percent or more of the saturation water content in the epoxy and polyamide composites. Measurements with polyimides (Kapton and HTS/PMR-15) indicate (ref. 16) that the intensity of the long-life component is too low to render this technique usable for such materials. (See fig. 5.) This may be the result of tighter polyimide structure, which inhibits positronium atom formation in them. Table II is a summary of the results (refs. 16 and 17) of positron long component lifetime changes for saturation water content in four different types of specimen.

The positron lifetime technique has been used to measure the moisture content of several experimental samples in order to verify its anticipated accuracy. The following example illustrates typical results attainable with this technique. An experimental Narmco 5208/T300 graphite-epoxy composite panel with 55 volume percent fiber was exposed to normal outdoors environment for several months. The panel was brought into the laboratory and several 1" × 1" test coupons were quickly cut from it. These coupons were quickly weighed in the as-received condition. Lifetime measurements were then made in two coupons in the as-received state. Other coupons were used to obtain the lifetime-versus-moisture-content calibration curve for the sample material. Using this calibration curve, the test-specimen moisture content was calculated to be 16 ± 7 percent of the saturation value as opposed to a directly measured value of 19.2 ± 0.6 percent. The experimental results are summarized in table III and shown in figure 6.

So far, the test samples dealt with have involved uniform moisture distributions. For such cases, continuous-energy positron-emitter radioactive

sources are perfectly acceptable. However, when the moisture distribution is nonuniform, the use of continuously tunable monoenergetic positron beams should make it possible to probe the nonuniform moisture content as a function of depth in the test materials. Positrons of well-defined energy come to rest and decay at the end of their range in the test material. Their lifetime thus reflects the fractional moisture content at the end of their range. By using continuously tunable monoenergetic positron beams, it should be possible to measure the moisture content at different depths in the sample. A technique for obtaining and using monoenergetic positron beams of variable energy is described in reference 19. It involves magnetic analysis of Na^{22} positrons before they are allowed to enter the test specimen. Calculations indicate that the positron energy spread $\Delta E/E$ for 2.54-cm-diameter targets located $\geq$20 cm from the center of the magnetic field is less than 10 percent, whereas the corresponding positron transit-time spread is $\leq$100 ps. These data suggest that it is possible to continue to refer the positron lifetime to the simultaneous (<10 ps) emission of the 1.28-MeV gamma ray following the positron emission.

CONCLUDING REMARKS

Positron lifetime in resins and fiber-reinforced resin-matrix composites has been shown to be a linearly decreasing function of the moisture content of the specimen, being minimum at the saturation moisture level. Thus, for uniformly distributed moisture in the specimen, radioactive source-derived continuous-energy positrons provide a useful means of nondestructively measuring its fractional moisture content. For specimens having nonuniform moisture distribution, it will be necessary to use magnetically analyzed positron beams to define the location where the positrons come to rest in order to measure the fractional moisture content there. By using continuously tunable energy monoenergetic positron beams, it should be possible to map the moisture depth distribution in the test specimen. Thus, it would appear that the positron lifetime technique is a viable method for nondestructively measuring the moisture content with potential for its depth distribution in polymeric substances.

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May 9, 1980

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TABLE I.- SOME CONSTANT RELATIVE HUMIDITY SALTS AND THEIR RESPECTIVE CHARACTERISTICS

Salt (chemical form)	Temperature of enclosure, °C	Relative humidity in enclosure, percent
$MgCl_2$	30 to 75	29 to 33
NaCl [a]	100	50
$Na_2Cr_2O_7 \cdot 2H_2O$	30 to 60	55
NH_4Cl	30 to 45	80

[a]Since moisture diffusion in resin matrices is a strong function of temperature, NaCl offers the most convenient quick means of introducing 50-percent saturation moisture content distributed uniformly in the test sample.

TABLE II.- SUMMARY OF POSITRON LIFETIME CHANGES FOR SATURATION MOISTURE CONTENT IN MOLECULAR SUBSTANCES

Material	Saturation moisture, weight percent	Change in lifetime with saturation moisture, percent
Narmco 5208 (pure resin)	5	12
HBRF-55A (pure resin)	4	11
Narmco 5208/T300 (62.5 volume percent fiber)	[a].9	8
HBRF-55A/Kevlar 49 (43 volume percent Kevlar fiber)	7	15

[a]The saturation moisture content of Narmco 5208/T300 is considerably lower than the value expected on the basis of the saturation moisture content in Narmco 5208 resin. This is probably caused by the different curing histories of the two samples tested.

TABLE III.- SUMMARY OF TEST RESULTS ON A NARMCO 5208/T300

COMPOSITE SAMPLE

(a) Positron lifetime values in test sample

Condition of specimen	Positron lifetime, ps (a)	Moisture content, percent of saturation value
As received (test)	1780 ± 35	16 ± 7 (calculated)[b]
Completely dry	1793 ± 20	0 (measured)
Fully saturated	1696 ± 21	100 (measured)

(b) Comparison of calculated and measured moisture content values

Moisture content calculated using positron lifetime data, weight percent	Directly measured moisture content, weight percent
0.52 ± 0.22[c]	0.62 ± 0.02
16 ± 7 percent of saturation value[d]	19.2 ± 0.6 percent of saturation value[d]

[a]It should be noted that the positron lifetime values shown in figure 3(d) are not identical to those shown in this table. This is due to the fact that the specimens used for these two tests came from different sources and had different curing histories.

[b]This value has been calculated using the following expression:

$$\left(\begin{array}{c}\text{Fractional moisture content of test} \\ \text{specimen in the as-received condition}\end{array}\right) = \left(\frac{\bar{\tau}_{dry} - \bar{\tau}_{test}}{\tau_{dry} - \tau_{sat}}\right)$$

where τ is the measured value of the lifetime of positrons and $\bar{\tau}$ is the mean value. The errors in the calculated value reflect the errors in the calibration data.

[c]Error in the calculated value reflects errors in the calibration data.

[d]The saturation moisture content of the sample material was 3.22 weight percent.

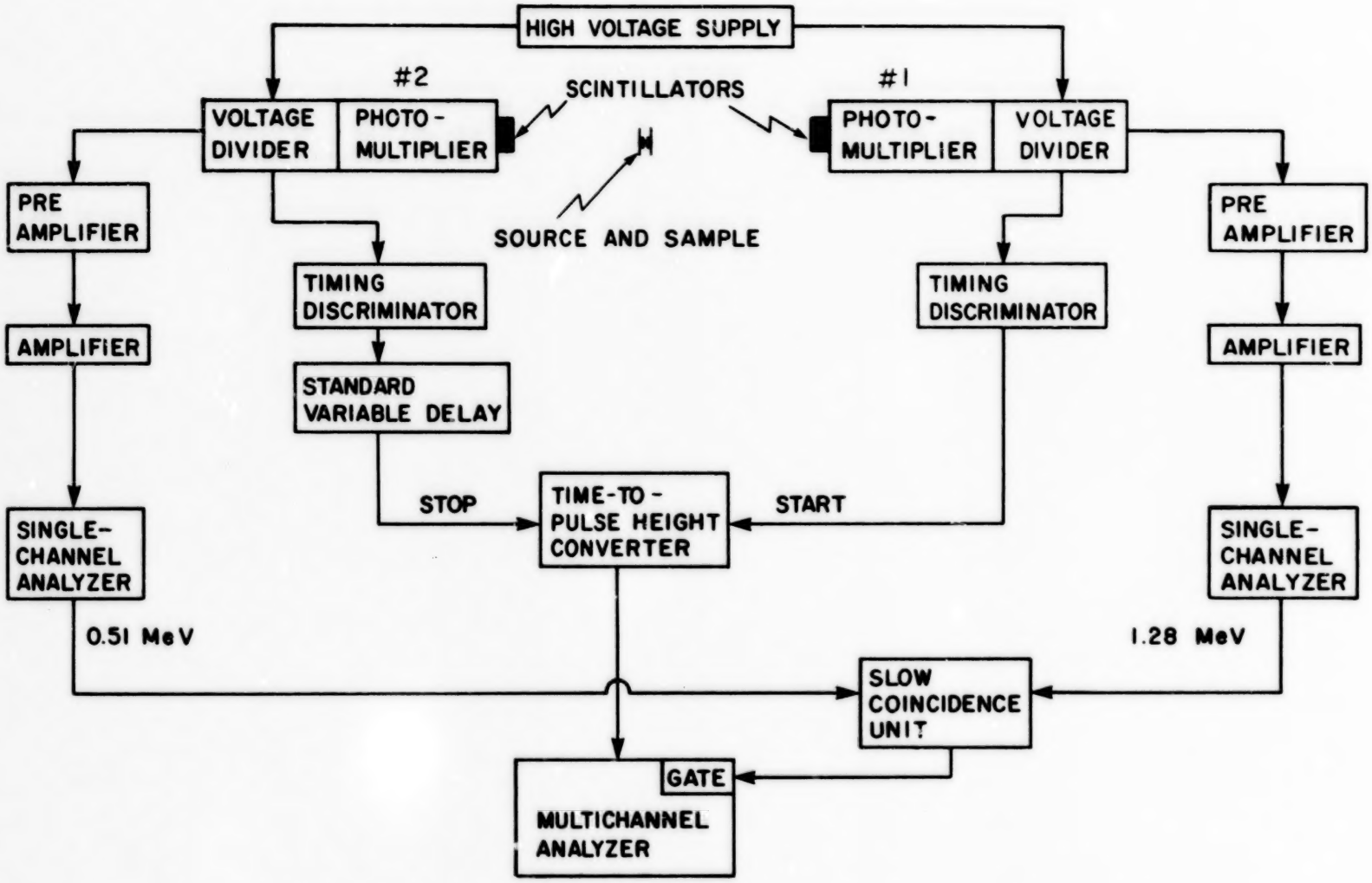


Figure 1.- Fast-slow coincidence system for measuring positron lifetime spectra.

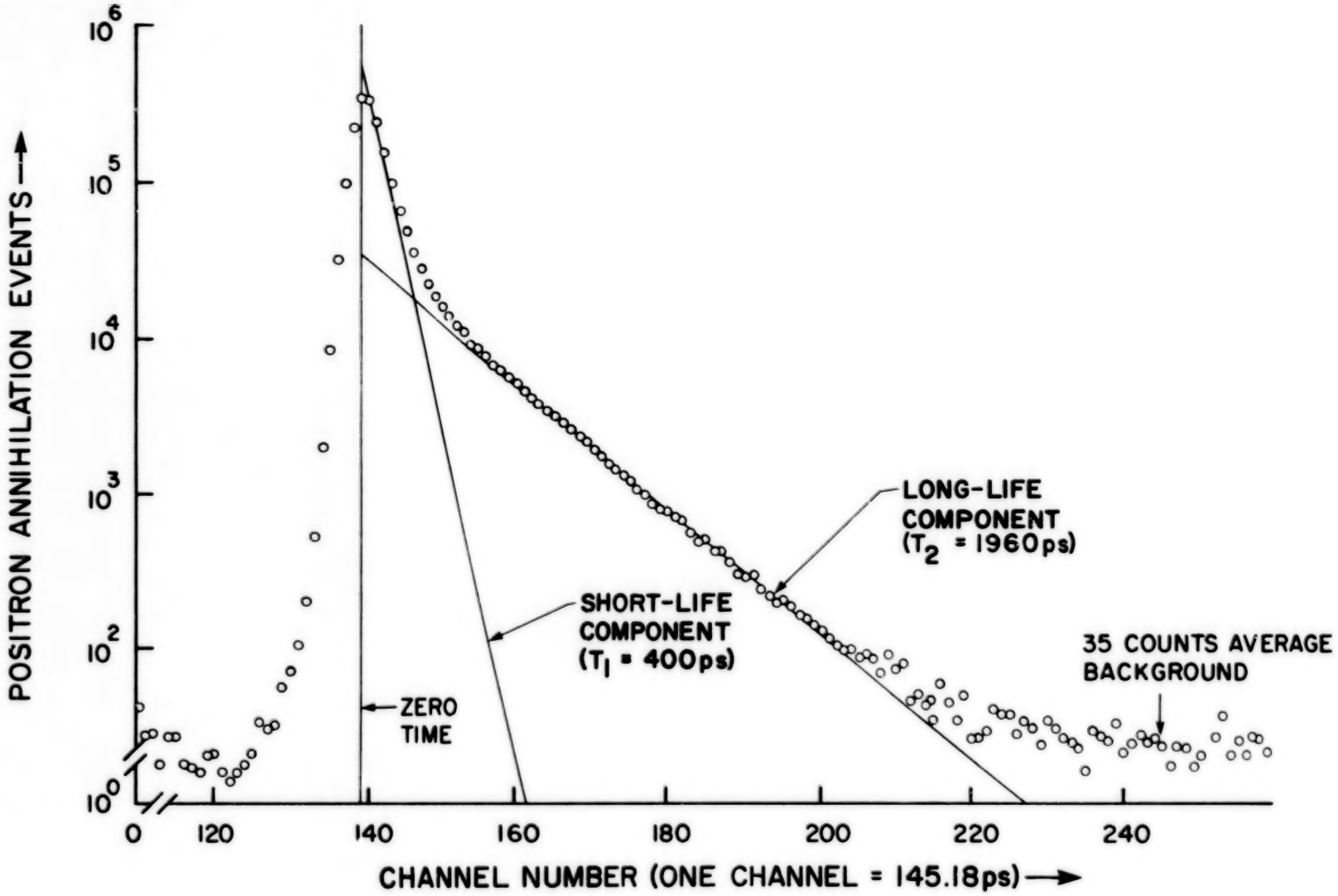
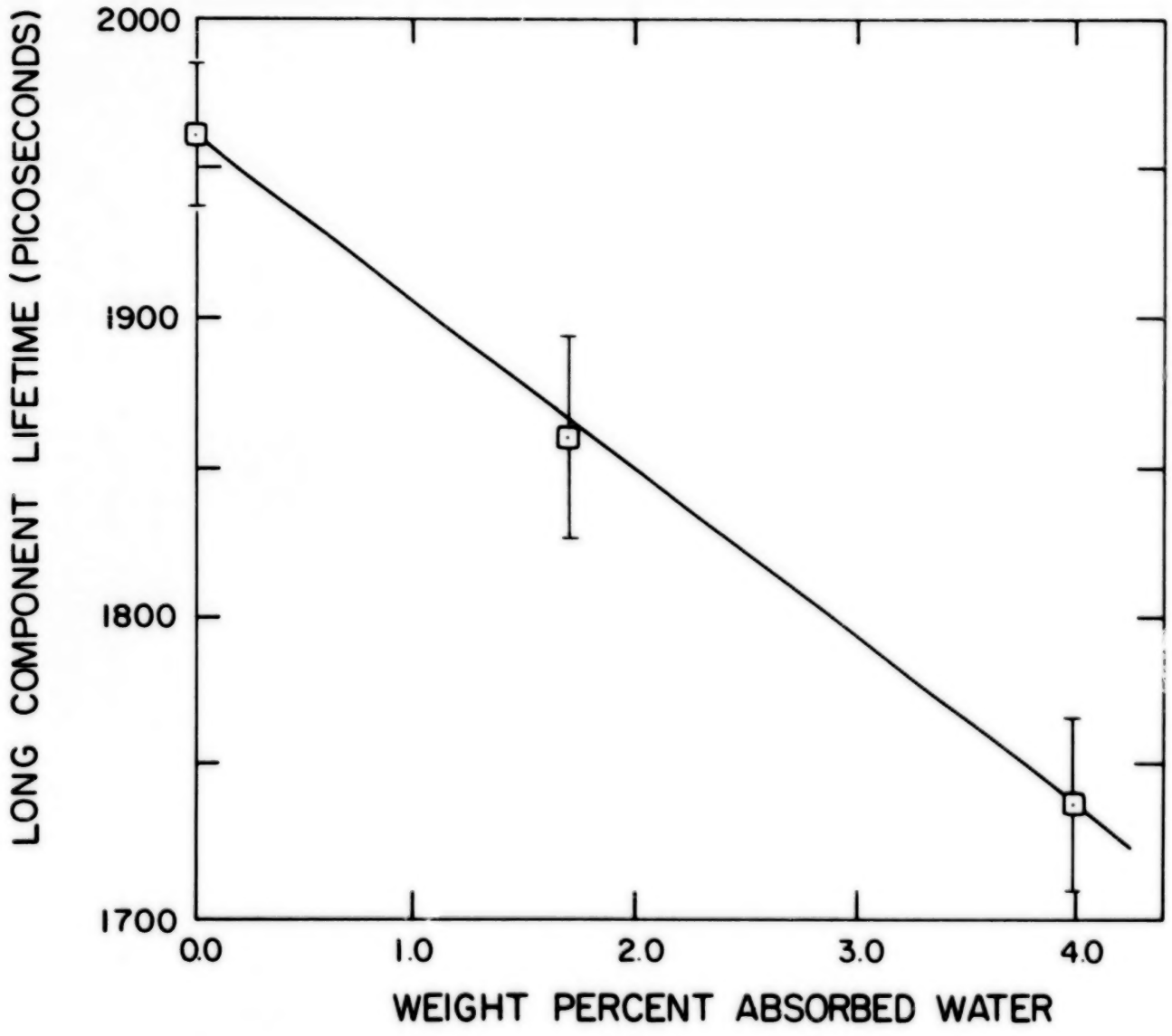
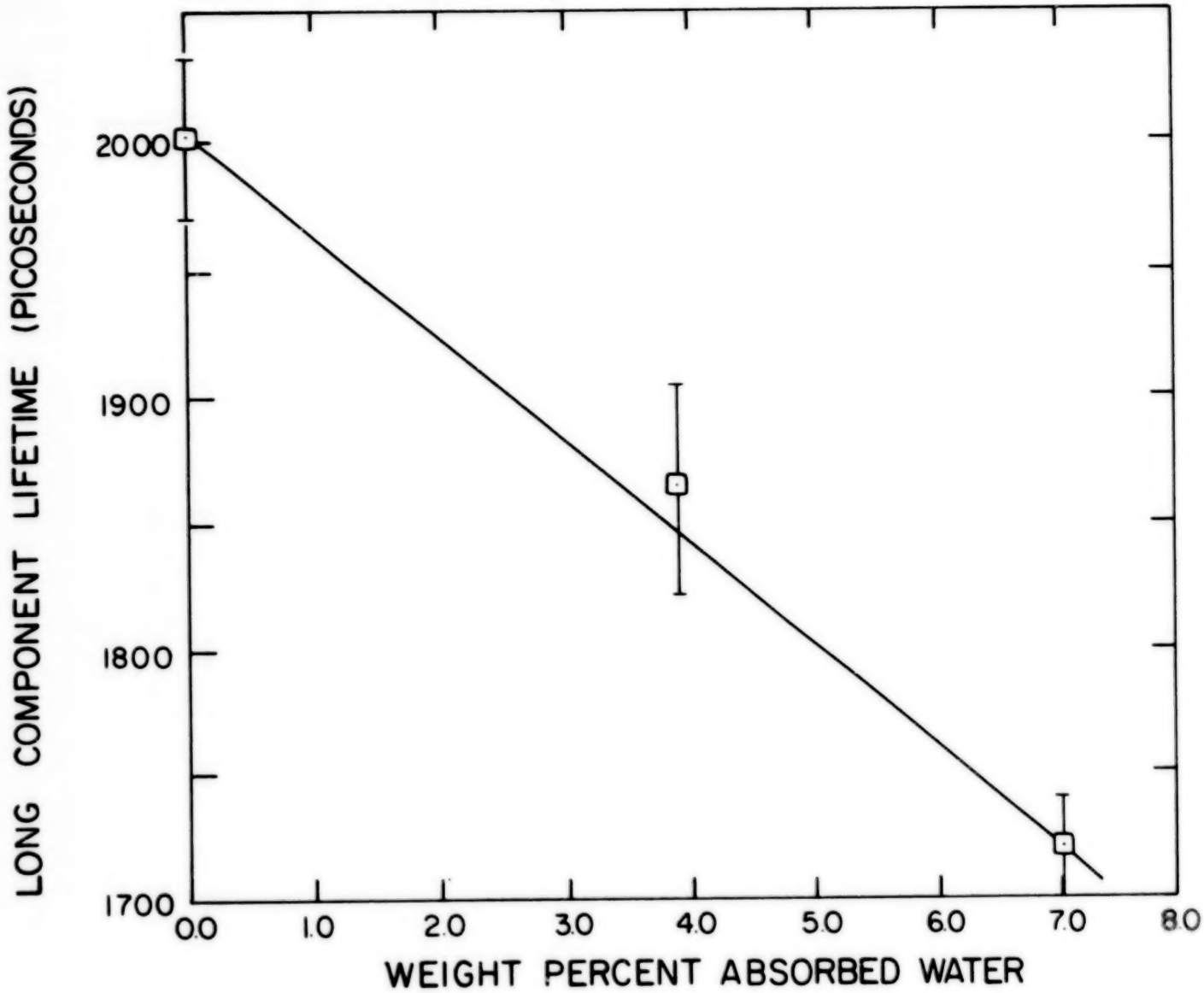


Figure 2.- Typical positron lifetime spectrum in a resin (HBRF-55A) specimen.



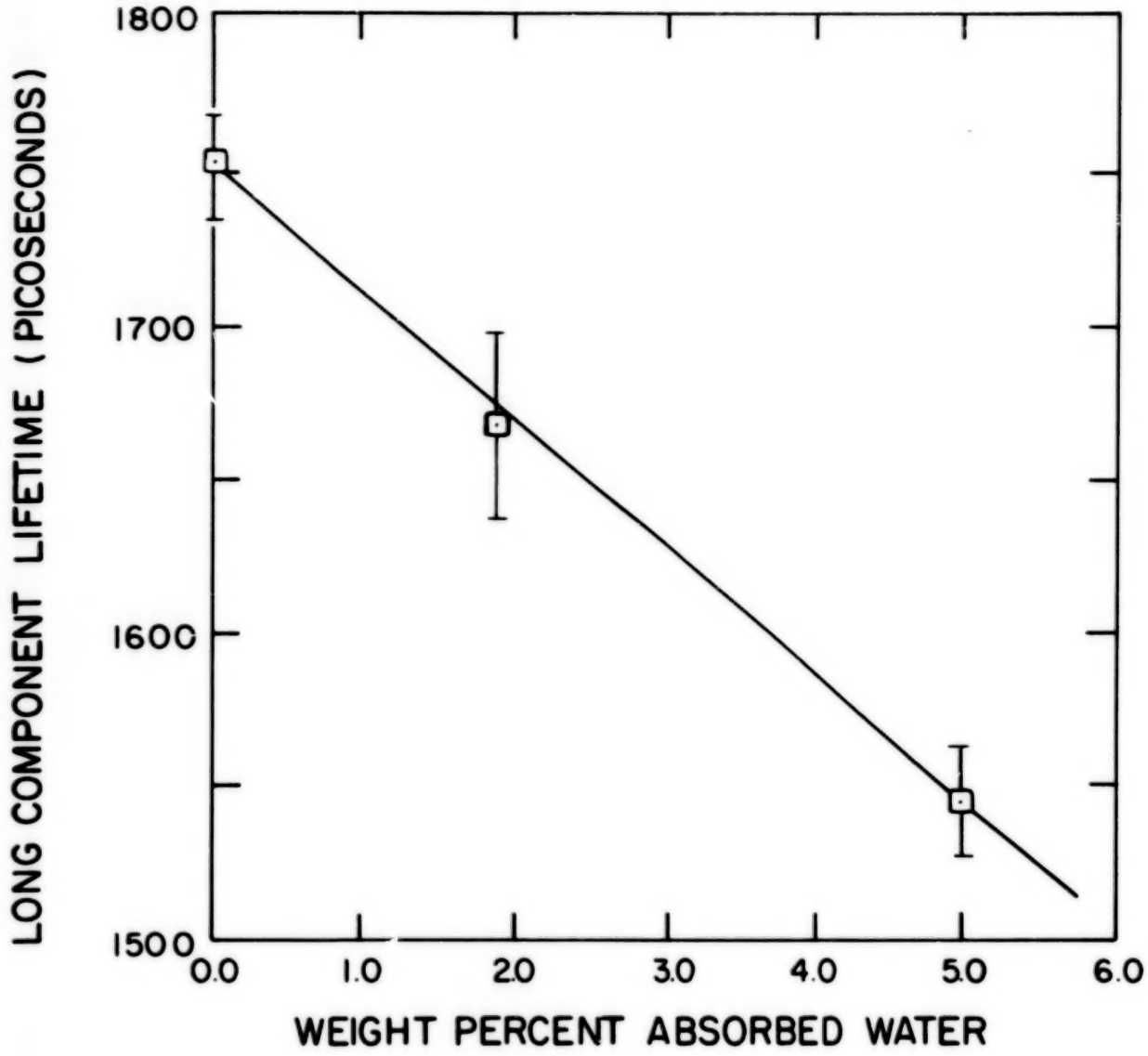
(a) HBRF-55A resin.

Figure 3.- Effect of moisture on positron lifetime.



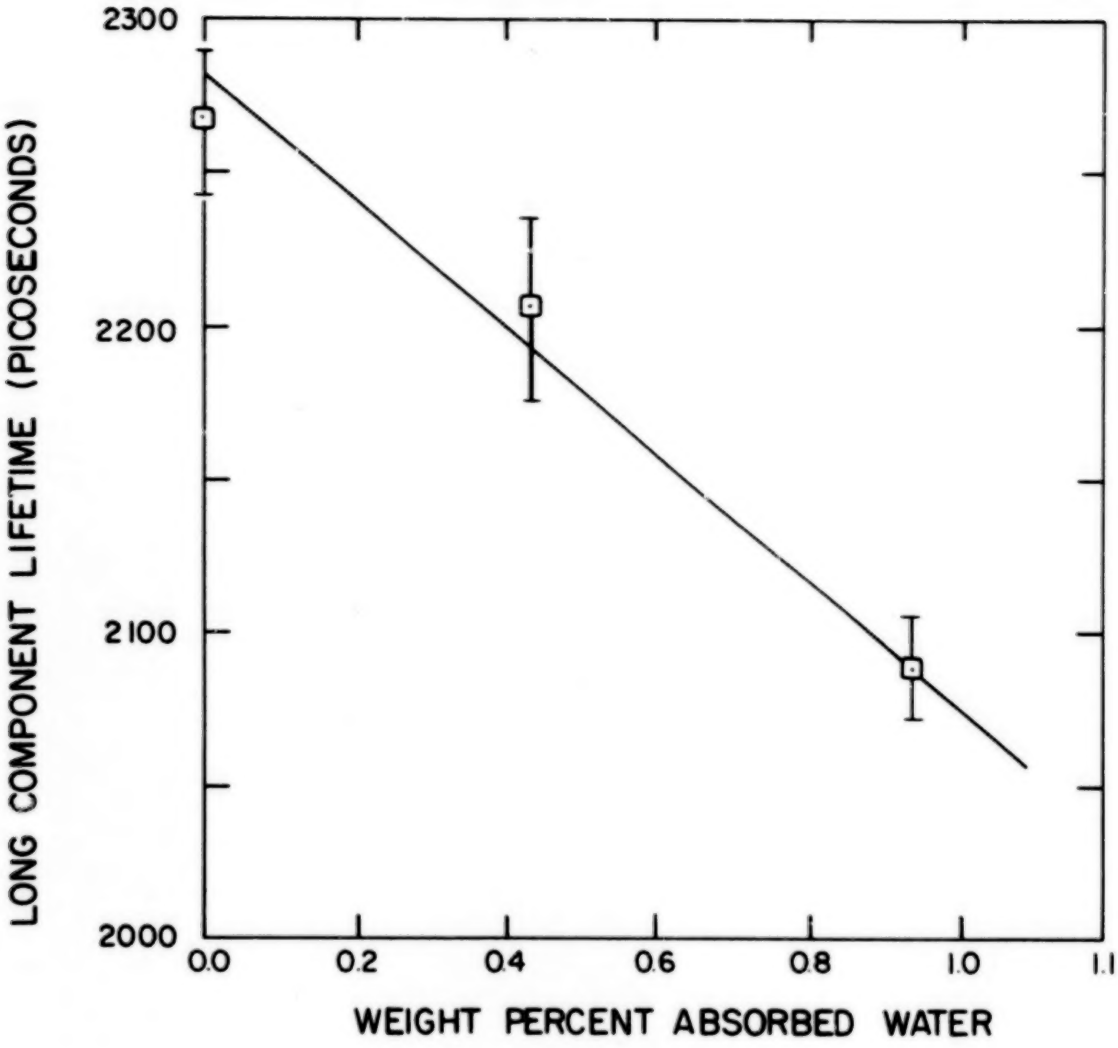
(b) HBRF-55A/Kevlar composite.

Figure 3.- Continued.



(c) Narmco 5208 resin.

Figure 3.- Continued.



(d) Narmco 5208/T300 composite.

Figure 3.- Concluded.

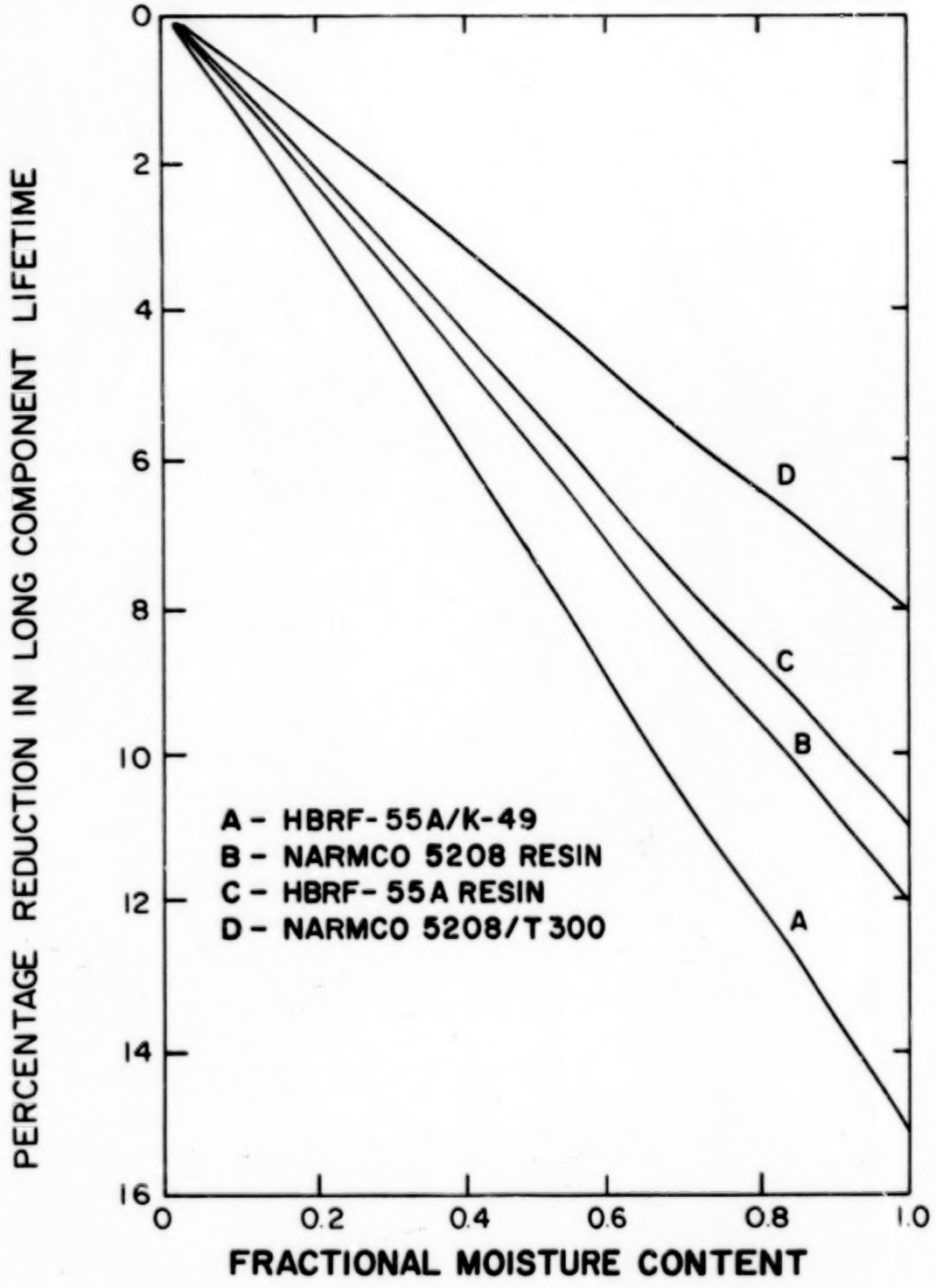


Figure 4.- Reduction in positron lifetime as a function of moisture content in selected resins and composites.

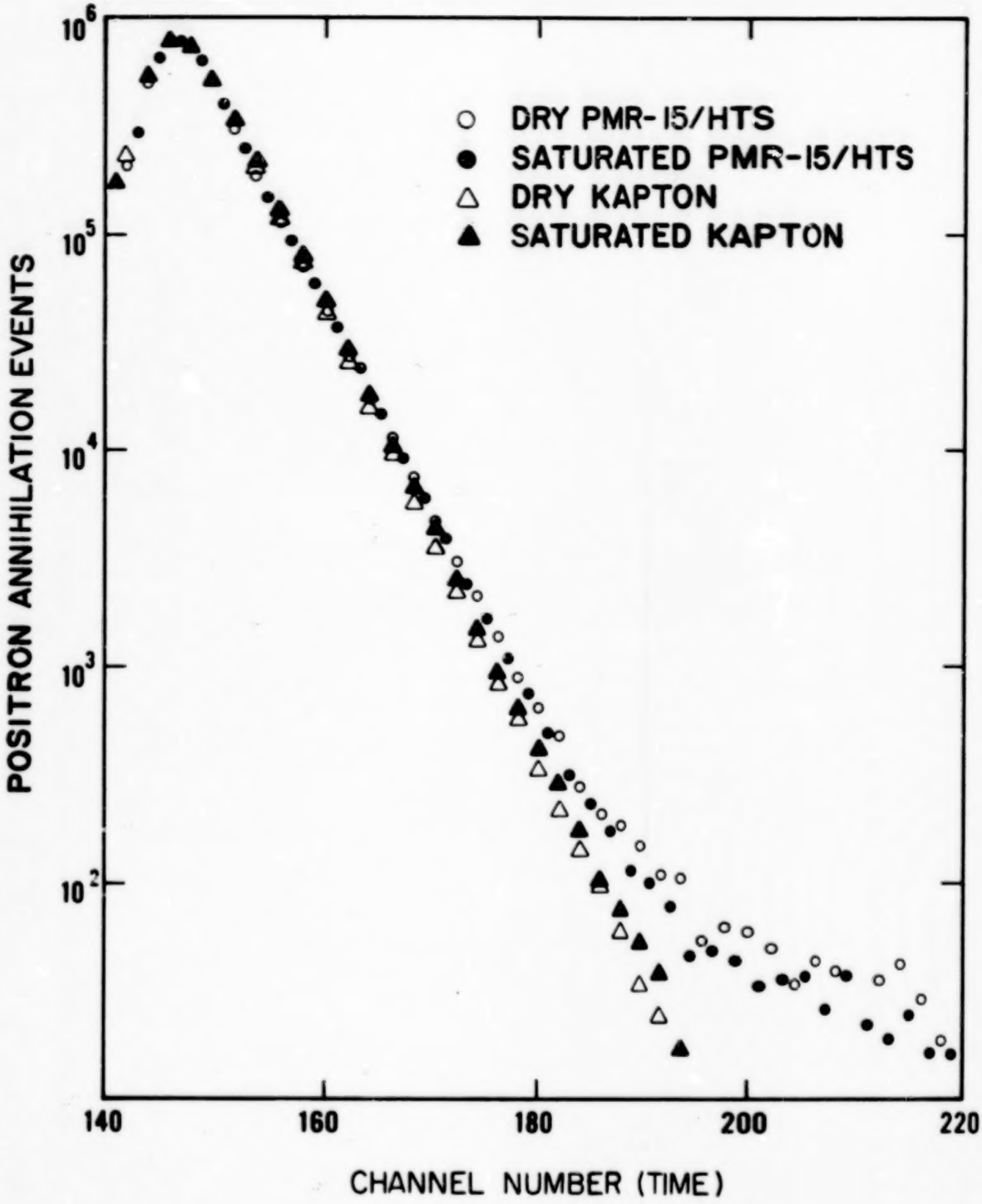


Figure 5.- Comparison of positron lifetime spectra for dry and wet polyimide-based materials.

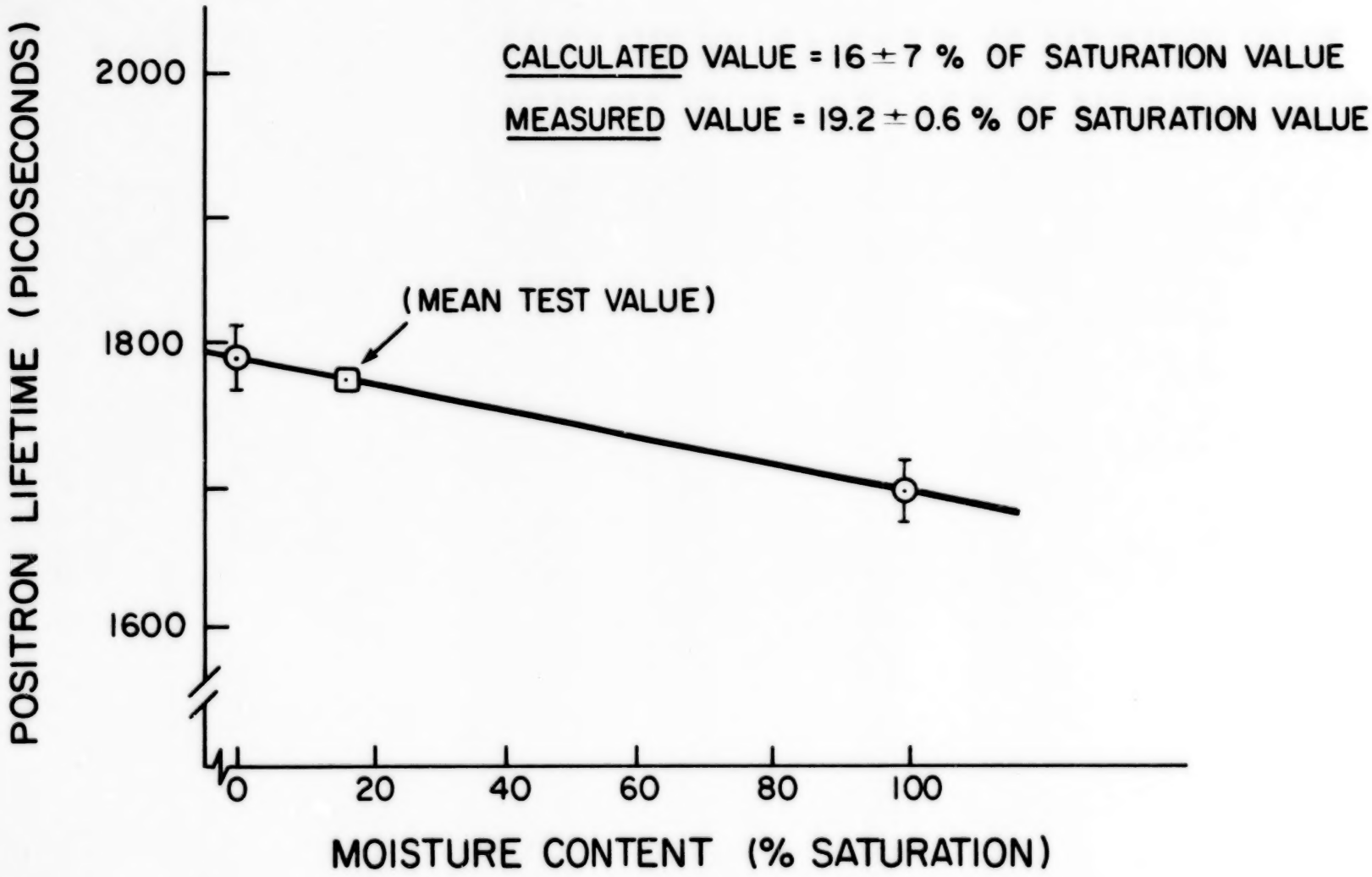


Figure 6.- Determination of moisture content in a test composite specimen (Narmco 5208/T300; 55 volume percent fiber).

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15. Supplementary Notes

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16. Abstract

Fiber-reinforced resin-matrix composites reportedly suffer significant degradation in their mechanical properties when they are exposed to hot, moist environments for extended periods. Moisture weakens the fiber-matrix bond as well as the matrix shear strength. Despite the importance of measuring moisture content and its effects on composite material properties, not enough data are available on suitable nondestructive techniques for detecting and measuring moisture diffusion in resin-matrix composite materials. This paper addresses the problem of measuring the moisture content of such materials, using the positron lifetime technique.

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